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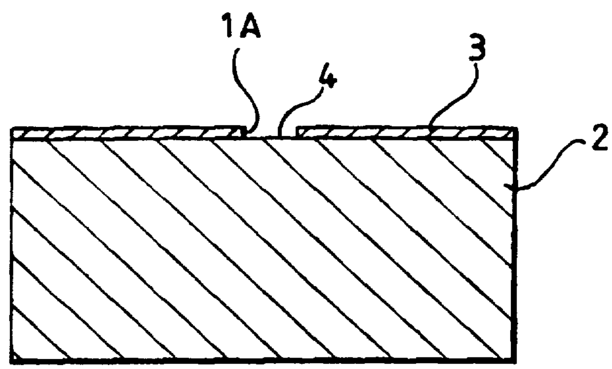
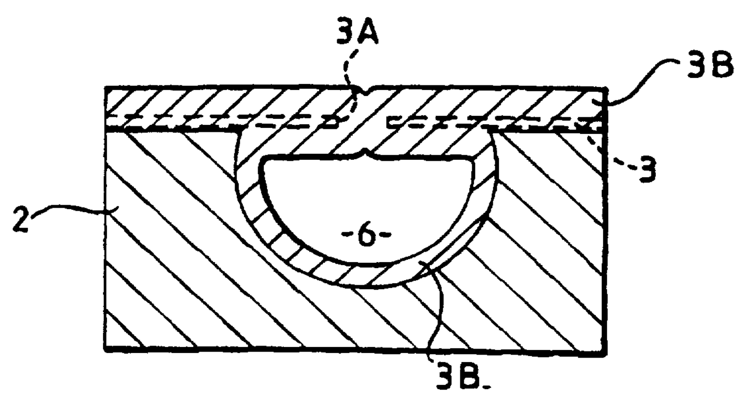
(54) Title: METHOD OF PRODUCING A HOLLOW STRUCTURE

(57) Abstract

A method is disclosed of producing a hollow structure, and particularly of forming channels of very small cross-sectional dimensions in a body of etchable material. In carrying out the method, a composite element is formed by providing a surface portion of a body (2) with a layer (3) of material resistant to etching medium for the body, the layer being interrupted to expose an area (4) of the body surface, etching medium is applied to the body at the interruption to etch a cavity (5) in the body beneath the resistant layer (3), and a further layer of material (3A, 3B) is provided on a surface of the composite element in the region of the interruption and/or the etched cavity. In a typical variant, the further layer is built up until it closes the interruption thereby forming a closed formation which may be an elongate tubular passage (6).





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METHOD OF PRODUCING A HOLLOW STRUCTURE

This invention relates to a method of producing a hollow structure from a body of etchable material and is particularly, although not exclusively, applicable to the formation of channels having very small cross-sectional dimensions, which may, for example, be in the sub-micron range, i.e. less than 10^{-6} metres and possibly as small as the nano range, i.e. of the order of 10^{-9} metres, in a solid body of etchable material, usually of metal. The invention also embraces hollow structures, including bodies having channels formed therein by the aforesaid method.

Cavities such as channels of relatively large cross-sectional dimension, which may be of the order of a millimetre for example, can be formed using conventional techniques such as investment casting, or electroforming around a disposable mandrel. Other known procedures for this purpose include the use of photolithographic techniques allied with electroforming to form a metal matrix around photo-resist images. All of these methods require the removal of support material, such as a mandrel or resist, after completion of the cavity formation and, whilst this is reasonably feasible for the aforesaid relatively large cross-sectional area cavities, it can become extremely difficult, if not impossible, at much smaller dimensions, such as in the sub-micron and nano ranges.

An object of the present invention is to provide a convenient practical method of producing a hollow structure from a body of etchable material and particularly of forming, in a solid body of such material, cavities such as channels having very small cross-sectional dimensions, which minimises or avoids the aforesaid problems.

According to the invention, a method of producing a hollow structure from a body of etchable material comprises forming a composite element by providing a surface portion of the body with a layer of material resistant to etching medium for the body, which layer is interrupted to expose an area of the body surface, applying etching medium to the body at said interruption to etch a cavity in the body beneath the resistant layer, and providing a further layer of material on a surface of the composite element in the region of the interruption and/or the etched cavity.

The further layer of material may be applied only to one or more edge surfaces of the resistant layer, or only to the surface of the cavity, or to a combination of at least parts of both these surfaces.

By continuing application of the further layer to at least the one or more edges of the resistant layer until the interruption therein is filled, it is possible to close the cavity. This can be particularly advantageous when the cavity is in the form of an elongate channel, the channel being conveniently closed to form a tubular passage by continuing application of the further layer to opposed edges of the resistant layer until the interruption therein is filled. Typically, the further layer is provided both over the opposed edges of the resistant layer and the surface of the cavity so that the further layer covers the entire surface of the closed channel.

The provision of the further layer, normally of metal, is preferably carried out by a deposition process such as plating. The resistant and further layers may conveniently consist of the same material.

The interruption in the resistant layer may include an area of larger dimensions than the remainder, which remains open after etching and the application of the further layer, whether the remainder of the interruption is closed or not, and the method then includes masking the wider area and applying material, which is the same as or different from that of the resistant layer, onto the already applied resistant layer to increase the thickness thereof, and removing the masking material to leave a connecting passage out of longitudinal alignment with, and preferably generally at right angles to, the tubular passage.

The invention will now be described, by way of example, with reference to the accompanying drawings in which:-

Figures 1 to 9 are diagrammatic representations of one form of the channel-forming method of the invention;

Figure 10 is a similar diagram illustrating an alternative form of the method;

Figures 11 and 12 are similar diagrams illustrating the formation of a double layer structure;

Figures 13 and 14 are similar diagrams showing another alternative form of the method of the invention;

Figure 15 illustrates part of a further alternative form of the method of the invention,

Figure 16 illustrates a development of the method of Figure 15.

The method of forming a hollow structure illustrated in Figures 1 to 10 of the drawings is initiated by providing a layer, indicated generally at 1, of resist material on a surface of a metal body or substrate 2, typically a blank copper sheet or wafer. The resist follows the line of a channel to be formed within the thickness of the sheet and will be seen from Figure 1 to include a central linear portion 1A defining the intended path of the channel to be formed and end portions 2A of larger area, intended for the formation of entry and exit ports for the channel, in the manner to be described. The type of resist employed is dependent upon the resolution required to create the desired initial image and compatibility with the chemical nature of the following stages. A very convenient resist is of the photo-resist type and, having been deposited using suitable masking, this is developed to fix it in position.

The next step is to coat the surface of the substrate carrying the resist by plating to form a layer 3 of nickel to a thickness of, for example, about 10 microns. This step is illustrated in Figure 2. The resist 1 is then removed as seen in Figure 3, forming a slot 1A through which a localised area 4 of the copper substrate is exposed through the layer 3 of nickel, the purpose of which latter, at this stage, is to act as a resist to an etchant to be used to create the channels in the copper substrate.

This etchant is now applied to the surface of the substrate and preferentially etches the copper over the exposed area 4, without having any etching effect upon the surrounding nickel coating 3. Figure 4 illustrates the substrate after etching has taken place and it will be seen that not only does etching occur into the depth of the substrate but also laterally beyond the confines of the slot 1A to produce a relatively wide generally semi-circular cavity 5 beneath the nickel layer 3, the

dimensions of the cavity being dependent upon control of the etching parameters.

Upon completion of the cavity, the substrate is subjected to a further metal deposition step as illustrated in Figure 5, conveniently employing nickel, over the entire exposed surface of the initially formed nickel layer 3, including the opposed edges 3A forming the gap 1A, and extending over the entire surface of the cavity. As the build-up of additional nickel 3B continues, deposition of nickel along the opposed edges 3A of the very narrow slot 1A finally fills the slot, completely blocking the top of the cavity and thereby creating a closed generally semi-circular channel 6 along the length of the slot 1A, although the two larger areas 2A (Figure 1) of the slot remain open. The completed channel is illustrated in Figure 6, the initial nickel layer 3 being represented in broken lines.

As can be seen from Figure 7 of the drawings, the channel 6 lying below the outer surface of the substrate 2 is connected to the two larger openings 2A formed within the thickness of the initially deposited nickel layer as a result of the shape of the resist material 1 initially applied to the copper substrate. With a deposit of nickel sufficient to close the slot 1A, the nickel layer 3 over the surface of the substrate would be insufficiently thick to enable ports to be formed at the locations 2A of sufficient mechanical strength to support the necessary external connections to be made to the assembly. It is therefore necessary to continue plating to increase the thickness of the layer 3, but at the same time to close off the openings 2A to prevent the channel 6 from being blocked by a build-up of nickel at the openings 2A. This is achieved by placing pads 7 over the openings 2A, preferably just before complete blockage of the slot occurs and prior to recommencing the plating

process which provides a substantial additional layer of nickel 8 on top of the originally deposited layer 3. It will be seen from Figure 8 that the layer 8 is several times thicker than the layer 3, so that upon removal of the masking pads 7 from each opening 2A, a port of significant depth is formed in communication with an end of the channel 6, permitting appropriate connections to be made to the channel for the supply thereto of fluid for example.

Figure 9 is a perspective view illustrating in more detail the substrate 2, initial deposited layer 3 and final thicker deposited layer 3B. Ports 6A are shown communicating respectively with the ends of the closed channel 6 formed within the thickness of the substrate 2. In the arrangement described the ports are mutually parallel and extend at right angles to the channel longitudinal axis, but different orientations of ports are possible, depending upon the intended use of the device.

As illustrated in Figure 10, it is possible to employ the method of the invention to provide an open channel 6 in the substrate 2. This is achieved by making the gap 1A in the initial resist 3 substantially wider than previously, so that a substantial degree of plating may be achieved within the cavity and over the exposed surfaces of the initial nickel layer 3, including the edges 3A, without closing over the top of the channel 6. Such an open channel may be useful as a guide for sub-division of the component after manufacture, or as a means of locating other components, such as gaskets, on the finished component.

Figures 11 and 12 illustrate the use of the invention in forming interconnected channels at two levels within a substrate. The method is carried out as previously described, using a substrate 10, to provide a

channel 11 extending beneath an initial nickel layer 12 and an additional nickel layer 13, the latter being provided, as already described, to increase the structural integrity of the device and to permit the formation of ports 14 connecting respectively with the ends of the channel 11. The ports 14 are formed with the aid of masking pads 15 of suitable resist similar to those designated 7 in Figure 8, but of substantially greater height than the pads 7. The next step is the deposition of a further substantial layer 16 of copper, or other suitable alternative material, being at least as thick as the original substrate 10 and forming a new base to accommodate a further longitudinal passage and end ports. These are created by repeating the sequence of operations described in connection with Figures 1 to 9, resulting in the production of a further nickel layer 17 (Figure 12) which is imaged, as previously described, to permit the etching of an additional channel 18 extending parallel with the original channel 11. It will be seen that the masking pads 15 extend to a sufficient height to enable the ports 14 to extend through the entire structure so as to provide communication between both passages 11 and 18 and the exterior. A multi-layer structure could be provided by repeating the process illustrated in Figures 11 and 12 as many times as required to create the desired number of layers. The interconnections between passages in the various layers may be varied as desired by varying the manner of use of the pads 15. It will be understood that the plurality of substrates may be of the same or different materials.

Although the channels illustrated in the foregoing description are of generally semi-circular cross-section, it is possible to produce channels of more nearly rectangular cross-section by encouraging the lateral progress of etching beneath the interrupted resist layer 3, 17. As can be seen in Figure 13, this can be achieved by providing an additional resist layer

19, of nickel for example, beneath the area of the copper layer 10 or 16 being etched, so that the etching can be allowed to proceed entirely through the copper substrate, where its progress will be arrested when it encounters the underlying nickel layer. Etching can then be allowed to continue laterally until the desire cross-section is achieved.

Figure 14 illustrates the use of a method embodying the aforesaid principle. A closed channel 20 is formed in a suitable metal substrate 21 using a nickel or other resist layer 22 having an interruption 23 along the intended line of the channel. The channel is etched and lined, in the manner described in connection with Figures 1 to 8, but an additional resist layer 24, as of nickel for example, is deposited on the opposite side of the substrate to that carrying the layer 22, thereby enabling etching to be effected through the entire thickness of the substrate 21 and to continue laterally in order to provide a generally more rectangular channel cross-section, as described above. This principle can clearly be applied in the formation of double and multiple layered structures of the kind previously referred to.

Although the principles of the invention have been described, for simplicity, in relation to the formation of a single linear channel at a particular level in a substrate, it will be understood that a non-linear channel may be envisaged and that a plurality of linear and/or non-linear channels may be formed simultaneously by using suitable masking of the basic substrate, so as to form networks of interconnecting channels at the same or different levels and similarly or differently directed within the substrate. Any convenient connections between channels may be formed at the same or different levels. The cross-sectional dimensions of the channels, which are governed to some extent by the thickness of the

deposited nickel layer within the initially etched cavities, can be readily controlled, within limits, by the chosen width of the slot in the first layer of resist, since plating of the cavity surface will continue in a regular and even manner until the slot in the resist is closed off. Again, although the method of the invention has been described by reference to the use of a copper substrate and associated nickel resist, it will be understood that a variety of metals may be used for the substrate and subsequent layers, consistent with the availability of a compatible resist layer to allow preferential etching of the substrate. Thus, for example, the method may be adapted for use with such metals as aluminium, gold, silver and others. The materials used to coat the basic substrate should conveniently be such as to be capable of chemical deposition, or at least capable of deposition by evaporation or sputtering techniques.

The possibility for creating extremely fine channels and ports in a thin metal plate by the method of the invention enables structures to be produced through which fluids may be readily introduced, enabling miniature heat exchangers to be envisaged useful for the cooling or heating of semi-conductors or small mechanical components, or in chemical processes for example. They may be used to construct small mechanical actuators, pumps, valves or sensors of very small dimensions, permitting the dosing and control of very small liquid volumes useful, for example, in the medical field, being small enough for implantation in the body.

The method of the invention may also be used to form self-supporting hollow structures, such as generally hemispherical cups, on an etchable substrate, by forming pits in the substrate, lining the pits with material acting as a resist to an etching medium for the substrate and

preferentially etching away the substrate from around the lined pits to leave the lining as a distinct structure supported on the remaining substrate. This may be achieved by imaging a surface of such a substrate, such as copper, so as to enable a layer of resistant material, such as nickel, to be deposited thereon having one or more areas of interruption in the form of small dots. This permits preferential etching of the substrate to take place so as to form an undercut pit of generally hemispherical shape in the substrate at the location of each dot. An electroplating or similar step is now carried out to deposit a layer of nickel over the internal surface of each etched pit to a thickness of typically $7\mu m$. Upon removal of the nickel resist from at least the surface areas of substrate around the pits, the substrate can be etched around the nickel deposits within the pits to leave self-supporting nickel cups connected to the underlying substrate. An example of such a structure is illustrated in Figure 15 in which the cup can be seen at 30 connected to the remaining substrate 31 by a stem 32.

In an extension of this method, a plurality of rows of pits may be formed in a substrate by imaging, nickel deposition and etching of the underlying substrate to form pits, as described previously. At this point however the layer of nickel resist is removed from the substrate and the substrate re-imaged with interconnecting lines at the same pitch as the pits. By subsequently plating the deposited image to form cups and etching around the formed cups, it is possible to form a structure consisting of a number of rows of hemispherical cups 30 interconnected by ribs or rods 33 of substrate remaining at the re-imaged areas. The method may be further extended to form a plurality of layers, each containing a plurality of cups.

It will be understood that this principle may be applied to structures other than the cups referred to. In particular, following the formation of a closed channel or open channel, as exemplified respectively in Figures 6, 7 and 14, or interconnected networks of such channels, as exemplified in Figure 12, selective etching of the substrate around the channels may be performed to leave self-supporting open or closed channels, or networks thereof which may remain connected to the substrate or not, as required.

CLAIMS

1. A method of producing a hollow structure from a body of etchable material, comprising forming a composite element by providing a surface portion of the body with a layer of material resistant to etching medium for the body, which layer is interrupted to expose an area of the body surface, applying etching medium to the body at said interruption to etch a cavity in the body beneath the resistant layer, and providing a further layer of material on a surface of the composite element in the region of the interruption and/or the etched cavity.

2. A method according to Claim 1, wherein the further layer of material is applied only to one or more edge surfaces of the resistant layer.

3. A method according to Claim 1, wherein the further layer of material is applied only to the surface of the cavity.

4. A method according to Claim 1, wherein the further layer of material is applied to one or more edge surfaces of the resistant layer and to the surface of the cavity.

5. A method according to any one of Claims 1, 2 and 4, wherein application of the further layer to at least the one or more edges of the resistant layer is continued until the interruption therein is filled and the cavity closed.

6. A method according to Claim 5, wherein the cavity is in the form of an elongate channel and the channel is closed to form a tubular

passage by continuing application of the further layer to opposed edges of the resistant layer until the interruption therein is filled.

7. A method according to any one of the preceding claims, wherein the further layer is metal provided by effecting a deposition process.

8. A method according to any one of the preceding claims, wherein the resistant and further layers consist of the same material.

9. A method according to any one of the preceding claims, wherein the interruption in the resistant layer includes at least one area of larger dimensions than the remainder which remains open after etching and the application of the further layer, whether the remainder of the interruption is closed or not, the method including masking each larger area and applying material onto the already applied resistant layer to increase the thickness thereof, and removing the masking material from each larger area to leave a connecting passage out of longitudinal alignment with the tubular passage.

10. A method according to Claim 9, wherein each connecting passage is arranged at right angles to the tubular passage.

11. A method according to any one of the preceding claims, wherein a second layer of substrate material is provided on said further layer, a further composite element is formed by providing a surface portion of the second layer with a layer of material resistant to etching medium for the second layer, the resistant layer being interrupted to expose a surface area of the second layer, applying etching material to

the second layer at the interruption in the resistant layer to etch a cavity in the second layer beneath that resistant layer, and providing an additional layer of material on a surface of the further composite element in the region of the interruption and/or etched cavity.

12. A method according to any one of Claims 1 to 10, wherein an additional resistant layer is provided on a surface of the body opposed to that carrying the resistant layer and etching is continued through the entire thickness of the body until arrested by the additional resistant layer.

13. A method according to Claim 7, wherein the interruption is in the form of a dot such that the etching step produces a localised pit, the further layer of material being provided over the surface of the pit, the resistant layer being then removed from at least the surface area of the body around the pit, and the body being further etched at this area to leave the further layer as a hollow structure supported on a remaining part of the body.

14. A hollow structure made by the method according to any one of the preceding claims.

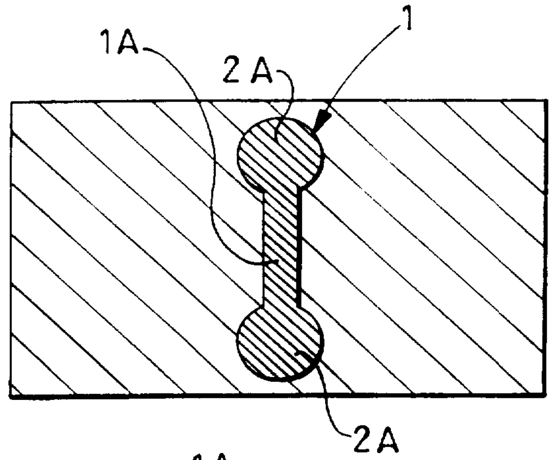

FIG 1

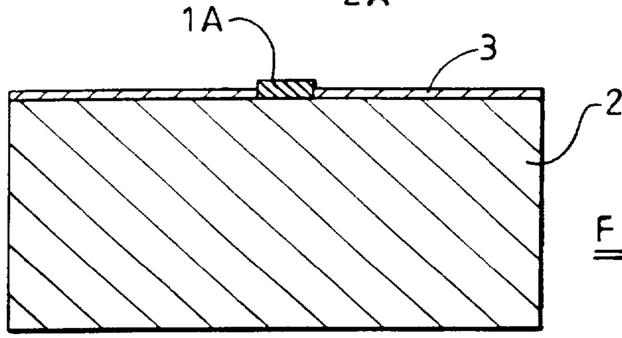

FIG 2

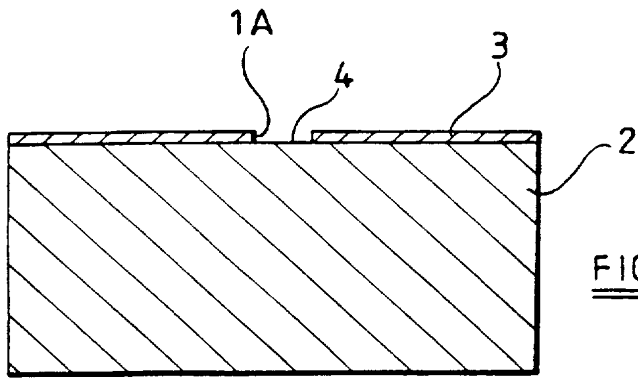

FIG 3

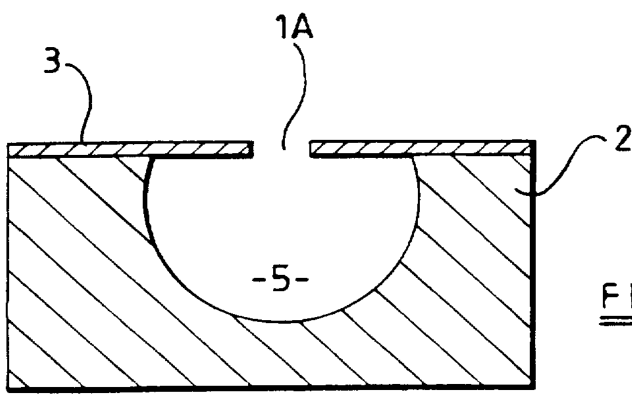

FIG 4

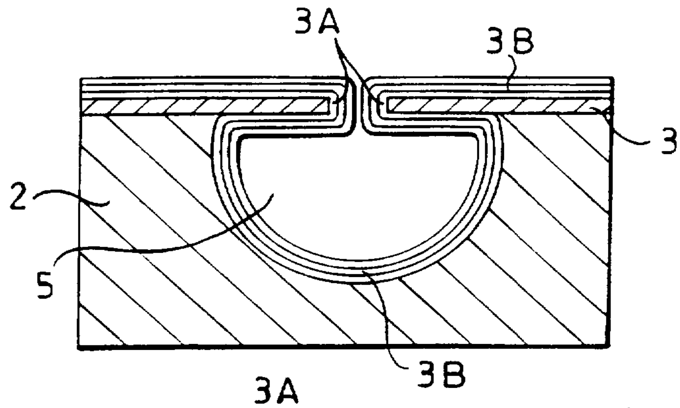

FIG 5

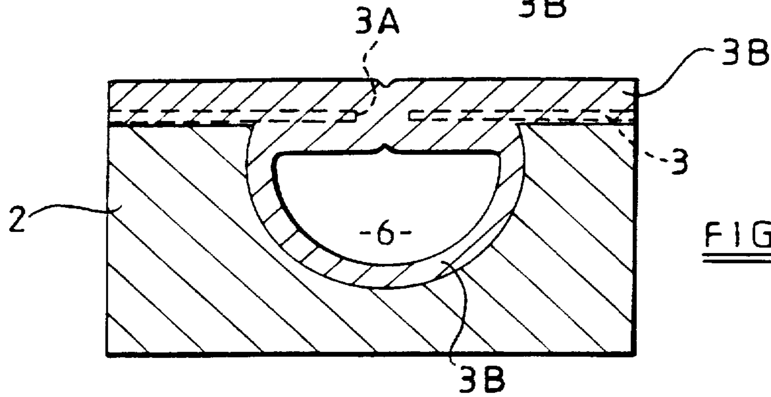

FIG 6

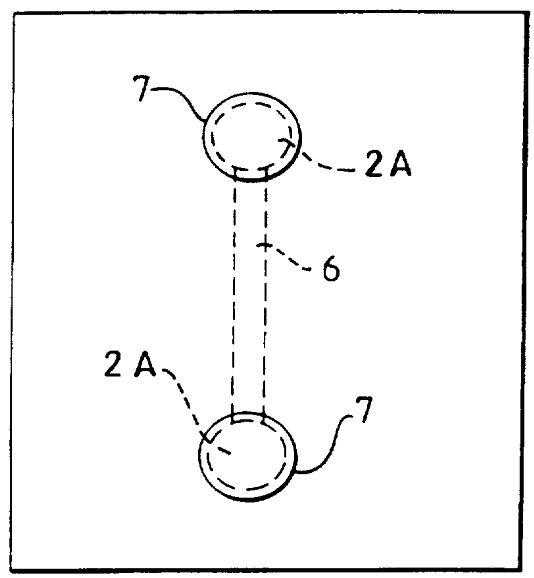

FIG 7

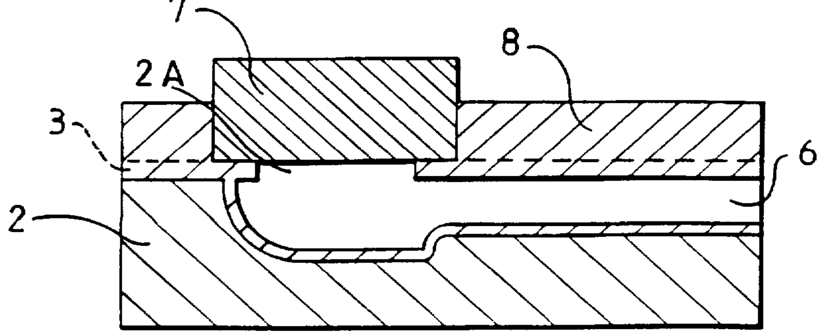

FIG 8

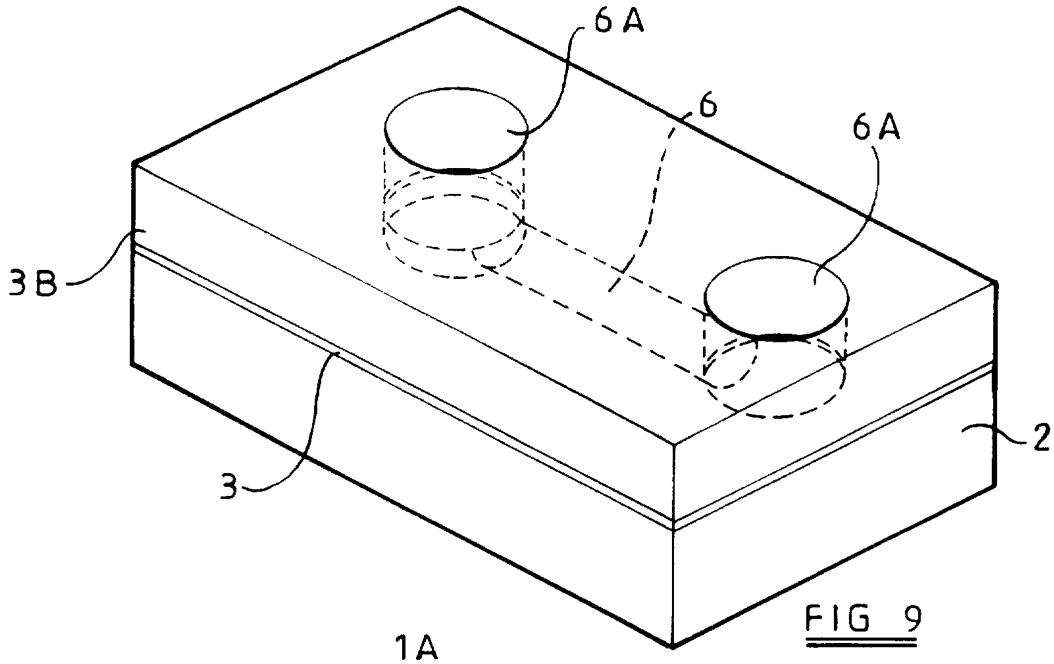

FIG 9

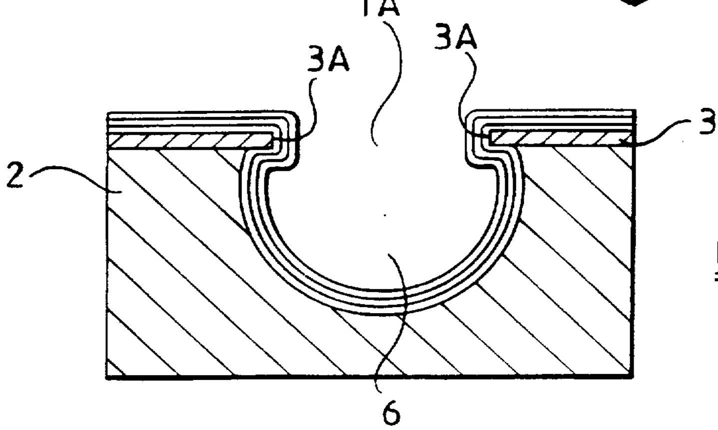

FIG 10

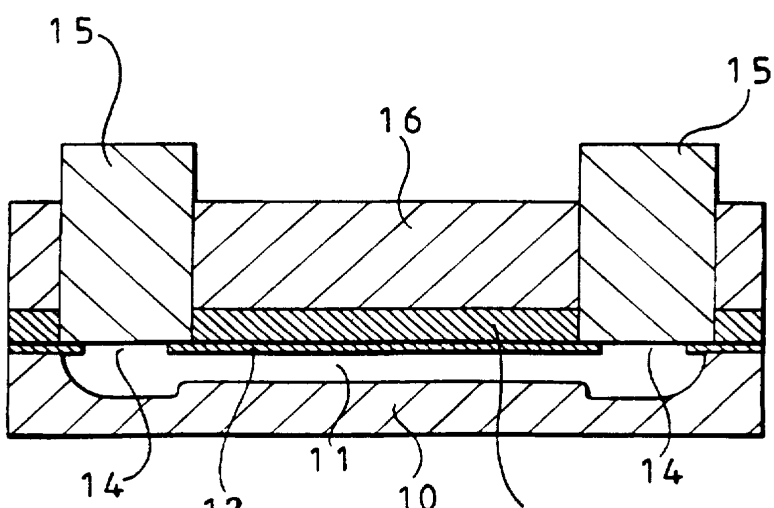

FIG 11

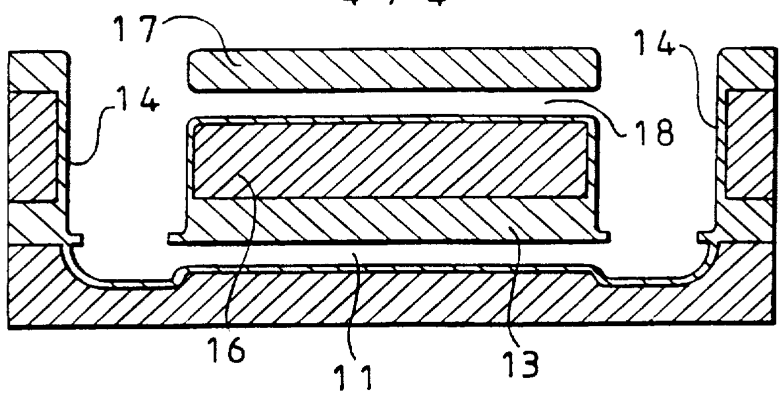

FIG 12

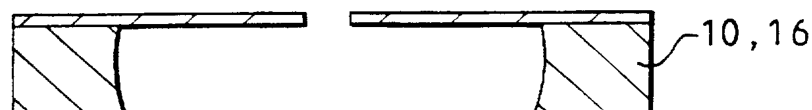

FIG 13

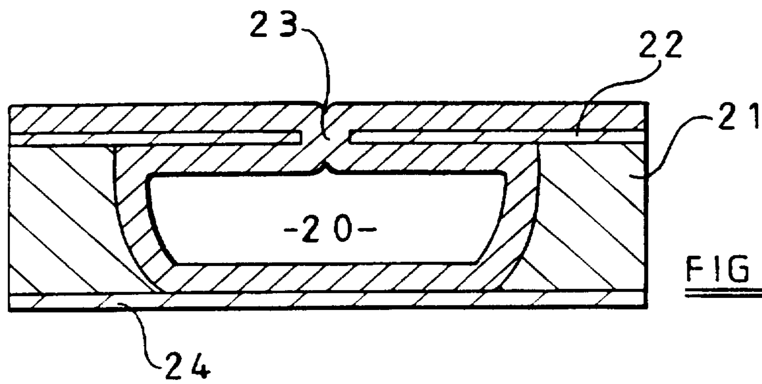

FIG 14

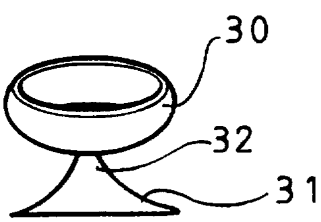

FIG 15

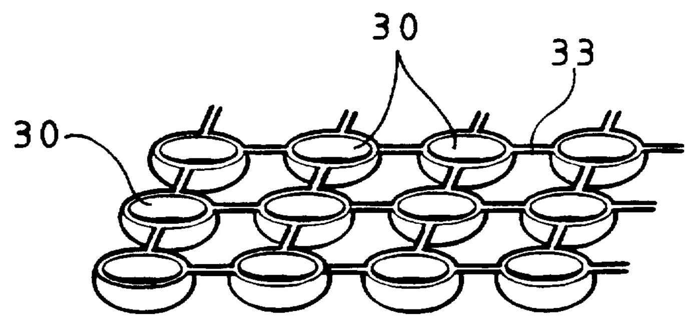

FIG 16

INTERNATIONAL SEARCH REPORT

Inte ional Application No
PCT/GB 97/02564

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C25D1/00 C23F1/02 C25D1/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C23F C25D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US RE34651 E (HOOPMAN TIMOTHY L ET AL) 28 June 1994 ---	
A	IBM TECHNICAL DISCLOSURE BULLETIN, vol. 26, no. 9, 1 February 1984, page 4785-4786 XP002050997 ---	
A	PATENT ABSTRACTS OF JAPAN vol. 012, no. 148 (C-493), 7 May 1988 & JP 62 263981 A (FUJITSU LTD), 16 November 1987, see abstract -----	

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☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US RE34651 E	28-06-94	US 4871623 A	03-10-89
		CA 1337184 A	03-10-95
		DE 68923105 D	27-07-95
		DE 68923105 T	25-01-96
		EP 0329340 A	23-08-89
		ES 2073431 T	16-08-95
		HK 167296 A	13-09-96
		JP 1655774 C	13-04-92
		JP 2022490 A	25-01-90
		JP 3022468 B	26-03-91
		KR 9615547 B	18-11-96